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<b>(21) International Application Number:</b> PCT/SE99/00627 <b>(22) International Filing Date:</b> 20 April 1999 (20.04.99) <b>(30) Priority Data:</b> 9801490-5                      28 April 1998 (28.04.98)                      SE <b>(71) Applicant (for all designated States except US):</b> SCA HYGIENE PRODUCTS AB [SE/SE]; S-405 03 Göteborg (SE). <b>(72) Inventor; and</b> <b>(75) Inventor/Applicant (for US only):</b> STRANDQVIST, Kersti [SE/SE]; Heymansväg 10, S-435 43 Pixbo (SE). <b>(74) Agent:</b> GÖTEBORGS PATENTBYRÅ DAHLS AB; Sjöporten 4, S-417 64 Göteborg (SE).		<b>(81) Designated States:</b> AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i> <i>In English translation (filed in Swedish).</i>
<b>(54) Title:</b> ABSORBENT STRUCTURE HAVING IMPROVED ABSORPTION PROPERTIES  <b>(57) Abstract</b>  An absorbent structure in an absorbent article such as a diaper, a pant diaper, an incontinence guard, a sanitary napkin or the like, said structure comprising a combination of a porous material such as fibers and/or foam, and at least 50 % by weight of a superabsorbent material in at least one area thereof in which the superabsorbent material is distributed. Said superabsorbent material is cross-linked by means of ionic bonds, preferably a polyvalent metal ion. The superabsorbent may further be covalently cross-linked.		

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**ABSORBENT STRUCTURE HAVING IMPROVED ABSORPTION PROPERTIES***Technical field*

5 The present invention refers to an absorbent structure in an absorbent article such as a diaper, a pant diaper, an incontinence guard, a sanitary napkin or the like, said structure comprising a combination of a porous material such as fibers and/or foam, and super-absorbent material. The invention also refers to an absorbent article containing such an absorbent structure.

10 *Background of the invention*

For absorbent articles of the above mentioned kind it is of essential importance that they have the ability to quickly receive and absorb large amounts of liquid. It is further of importance that liquid can be distributed from the wetting area to other parts of the absorbent structure, so that the total absorption capacity of the article can be utilized.

15

It is further essential that the absorbent structure can retain the liquid also when exerted to external loadings, such as when the user is moving, is sitting or lying down.

20 One problem, especially for diapers and incontinence guards, which are intended to receive and absorb relatively large amounts of liquids, is that they risk to leak before their total absorption capacity is fully utilized. A reason for such a leakage is that the absorbent structure, especially at repeated wettings, has a decreased capacity to quickly receive and absorb large amounts of liquid.

25 Absorbent articles of this kind comprise a liquid pervious topsheet, a liquid impervious backsheet and an absorbent structure arranged therebetween, said absorbent structure comprising a liquid acquisition layer located adjacent the liquid pervious topsheet, and one and more liquid storage and liquid distribution layers.

30 In order to obtain a high absorption capacity and a high liquid retaining capacity when the article is exerted to outer loadings it often contains so called superabsorbents. Superabsorbents are crosslinked polymers with capacity to absorb liquid in an amount

several times, 10 times or more, their own weight. They further have the capacity to retain absorbed liquid also when exerted to an external pressure. They have gained wide use in absorbent articles, at which they usually are in particulate form, such as grains, granules, flakes or fibers and they are mixed or layered with other absorption materials, usually cellulosic fibers.

The efficiency of a superabsorbent is dependant on many factors, such as where and how the superabsorbent is mixed into the absorbent structure, what physical shape the superabsorbent particles have, and the properties of the superabsorbent such as absorption rate, gel strength and liquid retaining capacity.

An important reason that the absorbent structure functions unsatisfactory at repeated wettings, i e at the second and third wetting occasions, is that it is difficult for the superabsorbent material to maintain its structure and shape after the superabsorbent particles have swollen. The firmness and shape of the superabsorbent particles can for example be weakened at outer loadings. By the fact that a superabsorbent at external loadings and after the first and second wetting has difficulty to maintain its structure and shape, a common phenomena called gelblocking occurs. Gelblocking means that the superabsorbent when wetted forms a gel which blocks the pores in the porous fiber structure and by that deteriorates the liquid transport from the wetting area to other parts of the absorbent structure. This involves that the total absorption capacity of the absorbent structure is not optimally utilized, and also involves an increased risk for leakage.

The problem of gelblocking increases when the amount of superabsorbent material in an absorbent structure is high. In order to make an article that is discrete and comfortable to wear it is however a desire that the article is thin. In order to maintain a high liquid absorption capacity such thin articles often have a relatively high amount of superabsorbent material.

In order to improve the capacity of the superabsorbent to maintain its structure even at an outer loading and after a plurality of wettings the superabsorbent material is often crosslinked in two steps. The first crosslinking is a so called internal crosslinking and is made by copolymerization of acrylic acid and at least one bifunctional agent under the formation of a network.

Copolymerizeable crosslinking agents used in superabsorbent polymers usually consist of bifunctional substances such as diacrylate esters and allylmethacrylates of trifunctional substances such as 1,1,1-trimethylol propane triacrylate and triallylamine or of tetra-functional substances such as tetraallyloxyethane.

The second crosslinking is a so called surface crosslinking and involves that the superabsorbent easier maintains its original shape also when exerted to external loads and after several wettings. Surface crosslinking of the superabsorbent is usually made by esterification of carboxylic groups. One example of surface crosslinking agents are polyhydroxy substances. Another example is organic carbonates, preferably ethylene carbonate in aqueous solution. A third example is the use of diglycidyl compounds, especially ethylene glycol-diglycidylether (EDGE).

It is also known through e g US 4,043,952 to surface crosslink a superabsorbent based on an anionic polyelectrolyte with a polyvalent metal ion, for example aluminum. The surface crosslinking occurs with ionic bonds. It is stated that the superabsorbent in question has an improved dispersability in an aqueous medium. There is no mentioning about an effect on the absorption capacity in an absorbent article.

Through EP-B-0 248 963 it is known to surface crosslink a superabsorbent of anionic character with a polyquaternary amine for increasing the absorption capacity of the superabsorbent. Also here the crosslinking is made by means of ionic bonding.

*The object and most important features of the invention*

The object of the present invention is to provide an absorbent structure in an absorbent article of the above mentioned kind and which contains a high amount of superabsor-

bent material, said absorbent structure having improved properties with respect to liquid acquisition capacity and distribution capacity at repeated wettings. This has according to the invention been achieved by the fact that the absorbent structure contains at least 50 % by weight superabsorbent material, based on the total weight of the structure in dry condition in at least one area thereof in which the superabsorbent material is distributed, said superabsorbent material is crosslinked by means of ionic bonds.

The superabsorbent material can also be covalently crosslinked by means of another crosslinking agent.

The superabsorbent material preferably comprises a polymer with anionic functional groups, for example a crosslinked polymer of a polyacrylate having functional carboxy groups.

The superabsorbent material is preferably crosslinked by cations, which by means of ionic bonds are bonded to the anionic functional groups of the superabsorbent material. According to one embodiment the cationic crosslinking agent comprises a polyvalent metal ion, for example aluminum, zirconium, chrome, titanium or zinc. According to a preferred embodiment the ionic crosslinking is made by aluminate ions,  $\text{Al}(\text{OH})_4^-$ .

The superabsorbent material can either be surface crosslinked by the ionic crosslinker or substantially homogeneously crosslinked therewith.

According to one embodiment the absorbent structure contains at least 70 % by weight superabsorbent material, based on the total weight of the structure in dry condition in at least one area in which the superabsorbent material is distributed.

The invention further refers to an absorbent article such as a diaper, a pant diaper, an incontinence guard, a sanitary napkin or the like comprising a liquid pervious topsheet, a liquid impervious backsheet and an absorbent structure arranged therebetween, at which the absorbent structure is of the kind stated above.

*Description of drawings*

The invention will in the following be closer described with reference to the accompanying drawings.

Fig. 1a and b show in the form of diagrams a comparison of the utilization degree of the absorption capacity of an absorbent article on different distances from the wetting area and where the article contains different superabsorbents in an amount of 50 and 70 % by weight respectively.

Fig. 2 a and b show in the form of bar charts the acquisition time at the first, second and third wetting occasions in absorbent articles containing different superabsorbents in an amount of 50 and 70 % by weight respectively.

Fig. 3 a and b shows in the form of diagrams the rewet after the first, second and third wetting occasions of absorbent articles containing different superabsorbents in an amount of 50 and 70 % by weight respectively.

Fig. 4 a and b show in the form of bar charts the momentaneous acquisition time under a certain load at the first, second, third and fourth wetting occasions in absorbent articles containing different superabsorbents with a content of 50 and 70 % by weight respectively.

*Description of embodiments*

Superabsorbent materials are defined as such materials that under gel formation can absorb many times their own weight, 10 times or more, of body liquids and test liquids comparable thereto such as synthetic urine or 0.9 % saline solution. Such materials may e g be hydrogel-forming polymers in the form of alkali salts of polyacrylic acid, polymethacrylic acid, copolymers of acrylic- and methacrylic acid with other monomers, acrylic acid grafted starch, polysaccharides such as carboxymethyl starch, carboxymethyl cellulose, xanthane, alginate, chitosane, pectine, guar gum, and peptides and proteins etc. The hydrogel forming polymers are usually crosslinked for making them water insoluble. Commonly used crosslinkers are ethylene glycol dimethacrylate, diethylene glycol diacrylate, allylmethacrylate, 1,1,1-trimethylolpropane triacrylate, triallylamine and tetraallyl oxyethane, which are bonded to the polymeric structure by means of covalent bonds.

According to the invention the superabsorbent is surface crosslinked or homogeneously crosslinked by a polyvalent crosslinking agent by means of ionic bonds. Besides it is preferably also covalently crosslinked by another crosslinking agent. The superabsorbent material preferably comprises a polymer with anionic functional groups such as carboxy groups, sulphonate groups, sulphate groups, phosphate groups, amide groups or nitrile groups, preferably however carboxy groups. The ionic crosslinking agent consists of cation, which by ionic bonds are bonded to the anionic functional groups of the superabsorbent material. The cation preferably comprises a polyvalent metal ion such as aluminum, zirconium, chrome, titanium or zinc.

Preferably the crosslinking agent is an aluminate ion,  $\text{Al}(\text{OH})_4^-$  in accordance with what is disclosed in the German patent application no. 198-18852.8 filed the same day. When producing such a superabsorbent aluminate ions are added to a water-containing gel of a carboxy group containing polymer, e.g. a polyacrylate, which preferably is crosslinked by means of covalent bonds in a conventional way. The gel is crosslinked with the aluminate ions before drying to a powder or granules, at which a homogeneously ionically crosslinked hydrogel structure is obtained.

When a superabsorbent absorbs liquid and swells the covalent bonds to the crosslinking agent are successively broken up, which leads to that the superabsorbent loses its gel stability in connection with the swelling. A superabsorbent however which is surface crosslinked or homogeneously crosslinked by a ionic crosslinking agent maintains its gel stability after swelling in a better way, due to that the ionic bonds can be rearranged and recreated as the polymer swells, which a covalent bond is not able to do.

The superabsorbent according to the present invention can thus be both covalently and ionically crosslinked, or only ionically crosslinked. The ionic crosslinking can either be on the surface of the superabsorbent particles or homogeneously through the entire structure of the superabsorbent.



The porous structure in which the superabsorbent is contained can be of optional kind, such as a fibrous structure of cellulosic fluff pulp, synthetic fibers of different kind, regenerated cellulosic fibers or mixtures thereof. The porous structure can also consist of an absorbent foam material.

5

The superabsorbent can either be mixed with the porous structure or be applied as a layer therein. It is also possible to apply a layer of superabsorbent material between two layers of porous material.

10 The amount of superabsorbent material in the absorbent structure should be at least 50 % by weight calculated on the weight of the structure in dry condition in at least one area in which the superabsorbent material is distributed. This means that in for example a double- or multilayered structure with different amounts of superabsorbent material in the different layers, the amount of superabsorbent material should be at least 50 % by  
15 weight in at least one of the layers. The total amount of superabsorbent material in the entire absorbent structure, however, could be lower than 50 % by weight.

According to one embodiment the amount of superabsorbent material in the absorbent structure should be at least 70 % by weight calculated on the weight of the structure in  
20 dry condition in at least one area in which the superabsorbent material is distributed

The physical shape of the superabsorbent material is important for its function in the absorbent structure and is preferably in the form of a powder, grains or granules.

25 Below a number of tests are disclosed which were made with respect to utilization degree, acquisition time and rewet in absorbent structures containing a combination of pulp fibers (chemical pulp) and 50 and 70 % by weight respectively of a superabsorbent material of four different types. The test absorbent bodies were of the size 28x10 cm, with a surface weight of 300 g/m<sup>2</sup> and a bulk of 3 cm<sup>3</sup>/g. The superabsorbent  
30 particles were substantially homogeneously mixed with the pulp fibers.

The superabsorbents that were used were a commercially available superabsorbent from Clariant GmbH named Sanwet IM 7100 and three different test substances of a polyacrylate homogeneously crosslinked by aluminate ions according to above and which besides was covalently crosslinked. The test substances were named E271/97, E214/97 and E222/97 and were supplied by Clariant GmbH and Clariant Corp.

#### *Utilization degree*

The absorbent bodies were placed with an inclination of 30° in order to imitate the placing of the diaper during use. The lower end was in contact with a liquid bath of synthetic urine (formula according to below) and was allowed to suck liquid during 60 minutes. The length of the wet area was measured. Test bodies were cut in smaller pieces corresponding to different lengths and were weighed. The utilization degree was calculated according to the formula:

Utilization degree = Absorbed weight of urine (g) / Theoretical absorption capacity (g),  
at which

Absorbed weight of urine = Weight of the sample after absorption - Dry weight;  
Theoretical absorption capacity = Weight of superabsorbent (g) in sample · Free swell capacity in synthetic urine + Weight pulp fibers (g) · Absorption capacity of pulp.

The absorption capacity for pulp fibers were measured after 60 minutes of free absorption in synthetic urine to be 8 g urine / g pulp.

Free swell capacity for the superabsorbent was measured by the below described method.

The result is shown in the diagrams in Fig. 1 a and b, from which it can be seen that the utilization degree at the portions located furthest away from the wetting area were clearly better for the test substances as compared to the reference.

*Formula for synthetic urine*

0.66 g/l magnesium sulphate, 4.47 g/l potassium chloride, 7.60 g/l sodium chloride,  
18.00 g/l urea, 3.54 g/l potassium dihydrogen phosphate, 0.745 g/l sodium hydrogen  
5 phosphate, 1.00 g/l 0.1-%-ig triton, 0.4 g/l Nykockin (colour), rest de-ionized water.

*Free swell capacity*

Pouches of polyester net 7x12 cm were prepared. 0.2 g of superabsorbent were weightd  
and placed in the pouches, which were welded and weighed. The pouches were  
10 immersed in synthetic urine during 60 minutes after which they were taken up, were  
allowed to drain and were weighed. The difference in weight after and before absorp-  
tion gives the free swell capacity.

*Acquisition time*

15 Three additions of each 80 ml synthetic urine were made through a plexiglass tube  
(inner diameter 23 mm) placed on the test bodies. The time interval between the  
additions were 10 minutes. The time it took until all liquid was absorbed was measured  
(visual observation). The result of the measurements is shown in Fig. 2 a and b. It is  
seen that the acquisition time at the second and especially the third addition was lower  
20 for the absorbent bodies containing the test substances as compared to the reference  
substance.

*Rewet*

Rewet was measured after 10 minutes after each addition by placing a filter paper on  
25 the wetting area and load it with a weight of 2550 g during 15 seconds. The filter paper  
was weighed before and after the loading and the rewet was calculated.

The result is shown in Fig. 3 a and b, at which it can be seen that the rewet after the first  
wetting occasion was substantially the same and very low both for the absorbent bodies  
30 containing test substances and the reference substance respectively. For the absorbent  
bodies containing 50 % superabsorbent (Fig. 1a) there was already after the second

addition observed a difference in rewet between the reference body and the test bodies, a difference that remained also after the third addition. For the absorbent bodies containing 70% superabsorbent (Fig. 3b) the rewet was the same also after the second addition, but after the third addition the difference between the test bodies and the reference body was significant, in such a way that the test bodies had a considerably lower rewet than the reference body.

*Momentaneous acquisition time under a certain pressure*

The tested absorbent bodies were clamped under a plexiglass disc, which had a circular opening. Four addition each of 80 ml synthetic urine were made through the circular opening via a funnel and a petri disc. The time interval between the additions were 10 minutes. The time it took until all liquid had been absorbed was measured (visual observation). The result is shown in Fig. 4 a and b. The acquisition time for the test substance (E-222/97) was lower at all additions as compared to the reference substance (IM 7100). The difference was biggest at the third and fourth addition. Similar results were obtained with respect to 50 as well as to 70 % by weight superabsorbent.

Besides the shown advantages with respect to improved absorption properties of absorbent structures containing ionically crosslinked superabsorbents the latter have a further important advantage, viz. that they have a higher resistance to mechanical stress that may occur when handling the superabsorbent particles in for example a diaper machine. A superabsorbent particle which is only covalently surface crosslinked and where the surface layer is damaged due to mechanical stress will lose much of its gel stability. A superabsorbent particle which is ionically crosslinked, either homogeneously or only on the surface, will substantially maintain its gel stability even after mechanical damages of parts of the surface layer. In a superabsorbent particle that is ionically surface crosslinked the ionic crosslinking bonds can be redistributed over the particle surface and in such a way repair possible damages in the surface layer.

**Claims**

1. An absorbent structure in an absorbent article such as a diaper, a pant diaper, an incontinence guard, a sanitary napkin or the like, said structure comprising a combination of a porous material such as fibers and/or foam, and superabsorbent material,  
5 characterized in,  
that the absorbent structure contains at least 50 % by weight superabsorbent material, based on the total weight of the structure in dry condition in at least one area thereof in which the superabsorbent material is distributed, said superabsorbent material is cross-linked by means of ionic bonds.  
10
2. An absorbent structure as claimed in claim 1,  
characterized in,  
15 that the superabsorbent material also is covalently crosslinked.
3. An absorbent structure as claimed in claim 1 or 2,  
characterized in,  
that the superabsorbent material comprises a polymer with anionic functional groups.  
20
4. An absorbent structure as claimed in claim 3,  
characterized in,  
that the superabsorbent material is a crosslinked polymer of a polyacrylate having functional carboxy groups.  
25
5. An absorbent structure as claimed in any of the preceding claims,  
characterized in,  
that the superabsorbent material is crosslinked by cations which by means of ionic bonds are bonded to the anionic functional groups of the superabsorbent material.  
30
6. An absorbent structure as claimed in any of the preceding claims,  
characterized in,

that the cationic crosslinking agent comprises a polyvalent metal ion.

7. An absorbent structure as claimed in any of the preceding claims,  
characterized in,

5 that the metal ion is aluminum, zirconium, chrome, titanium or zinc.

8. An absorbent structure as claimed in any of the preceding claims,  
characterized in,

that the crosslinking agent is an aluminate ion,  $\text{Al}(\text{OH})_4^-$ .

10

9. An absorbent structure as claimed in any of the preceding claims,  
characterized in,

that the superabsorbent material is surface crosslinked by the ionic crosslinking agent.

15

10. An absorbent structure as claimed in any of the preceding claims,  
characterized in,

that the superabsorbent material is substantially homogeneously crosslinked with the  
ionic crosslinking agent.

20

11. An absorbent structure as claimed in any of the preceding claims,  
characterized in,

that the absorbent structure contains at least 70 % by weight of superabsorbent material,  
calculated on the total weight of the structure in dry condition in at least one area  
thereof in which the superabsorbent material is distributed.

25

12. An absorbent structure as claimed in any of the preceding claims,  
characterized in,

that the superabsorbent material is in the form of a powder, grains or granules.

13. An absorbent article such as a diaper, a pant diaper, an incontinence guard, a sanitary napkin or the like of the kind comprising a liquid pervious topsheet, a liquid impervious backsheet and an absorbent structure enclosed therebetween, characterized in,
- 5 that the absorbent structure is of the kind stated in any of the claims 1-12.

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## 50 Weight% Superabsorbent

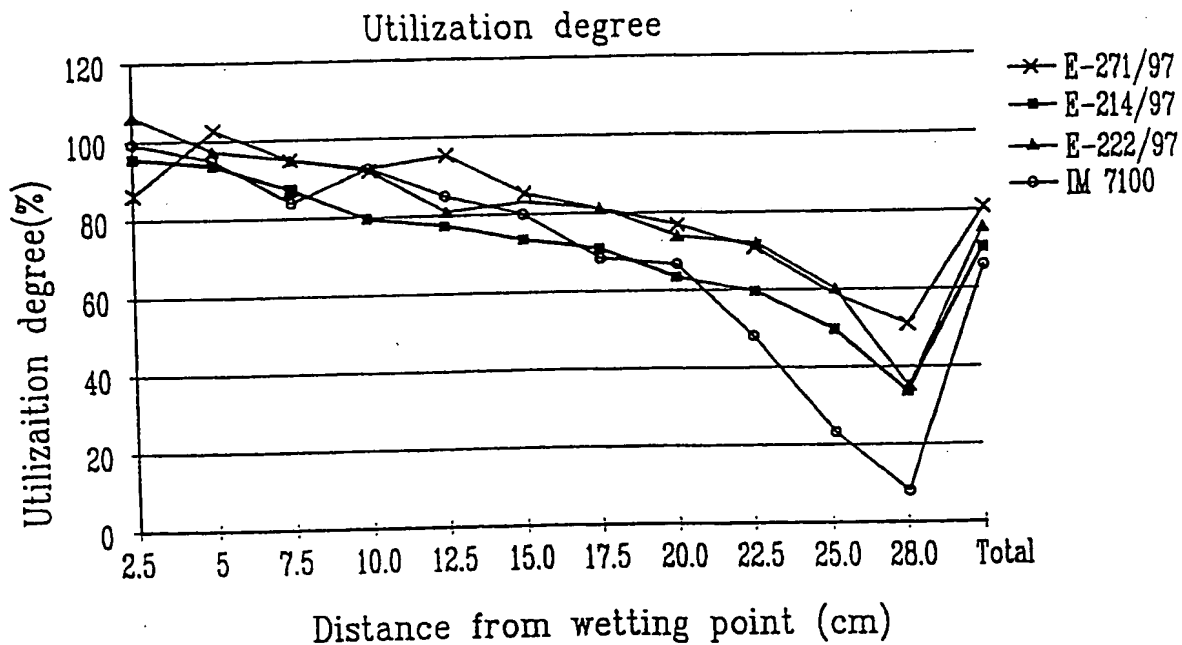


FIG.1a

## 70 Weight% Superabsorbent

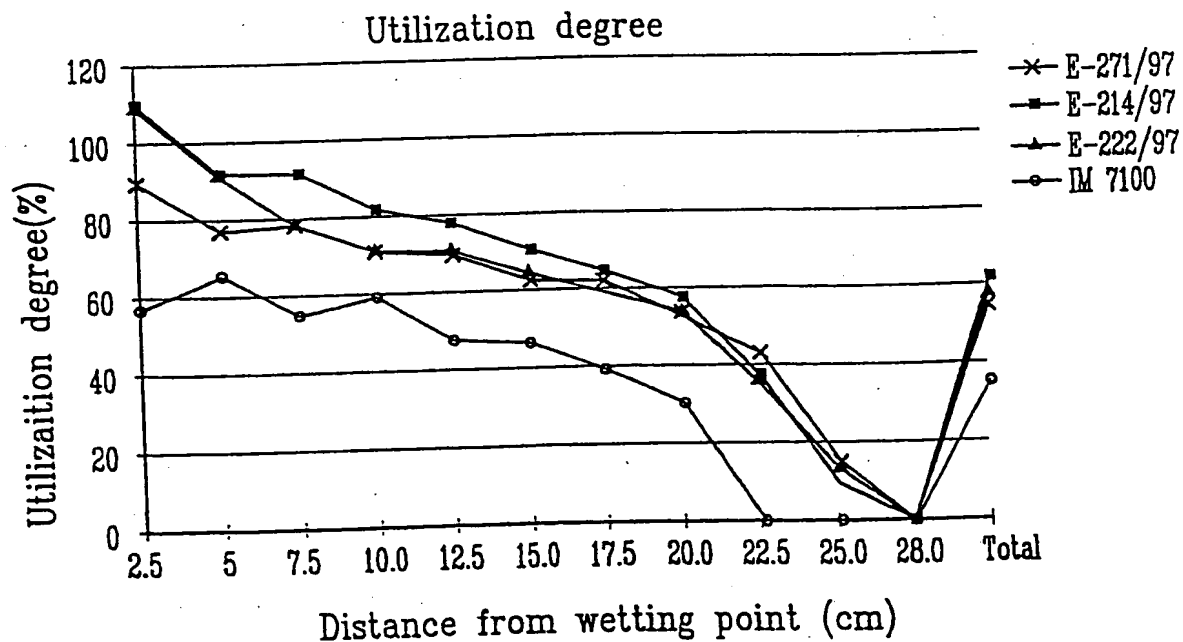


FIG.1b



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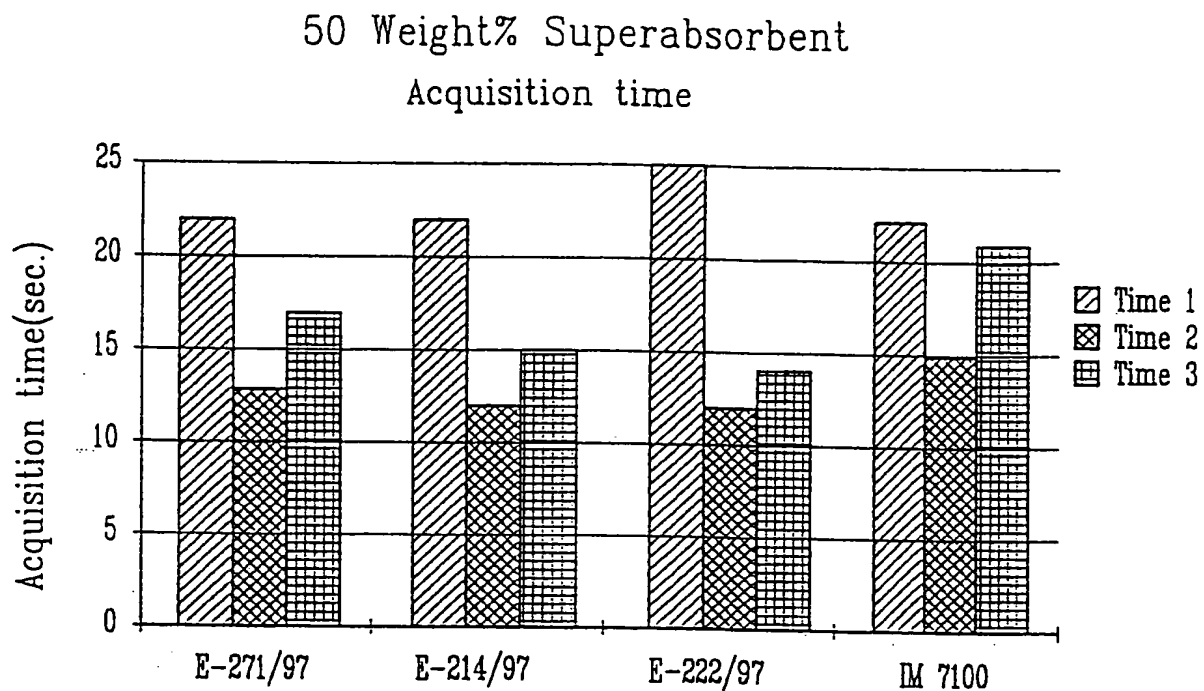


FIG.2a

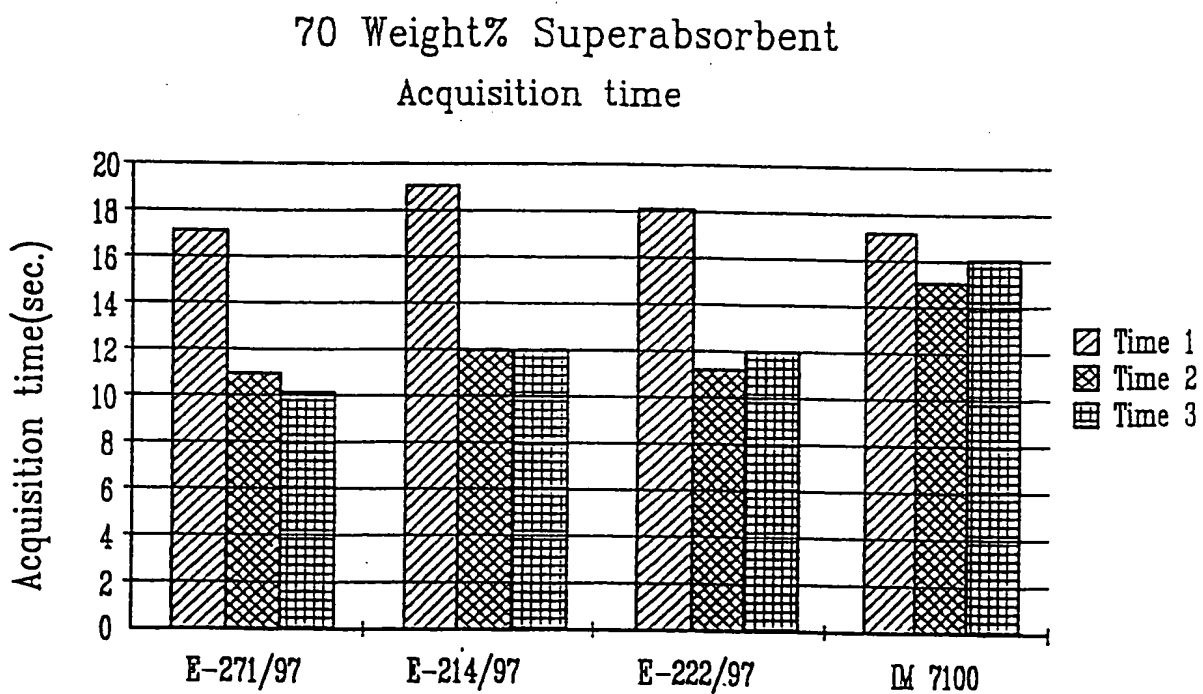


FIG.2b

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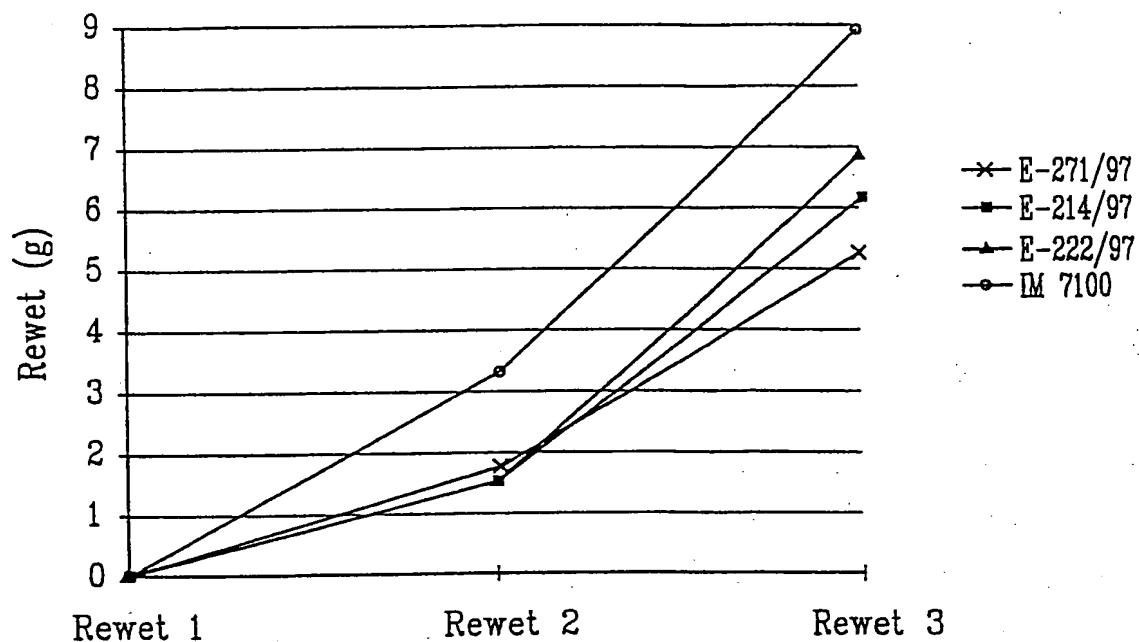
50 Weight% Superabsorbent  
Rewet

FIG.3a

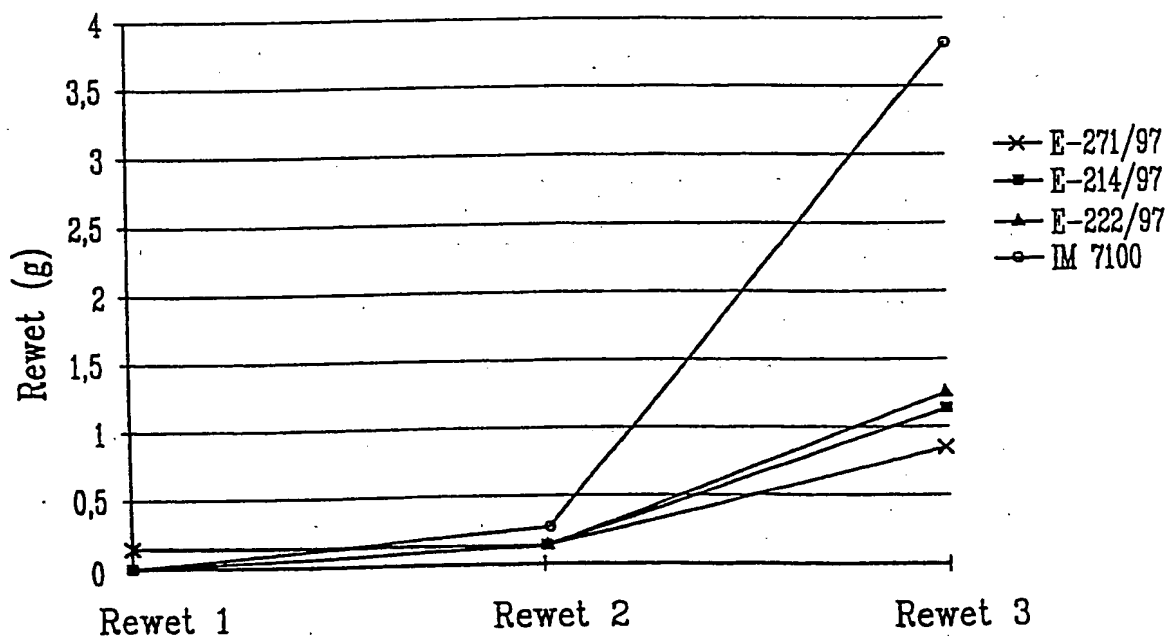
70 Weight% Superabsorbent  
Rewet

FIG.3b

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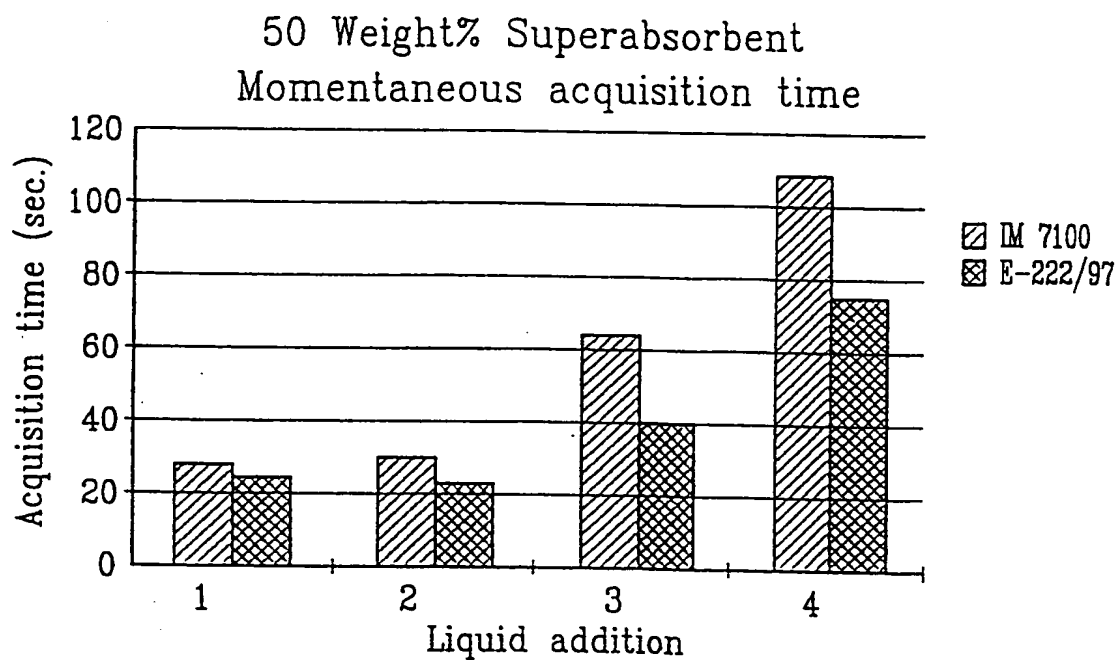


FIG.4a

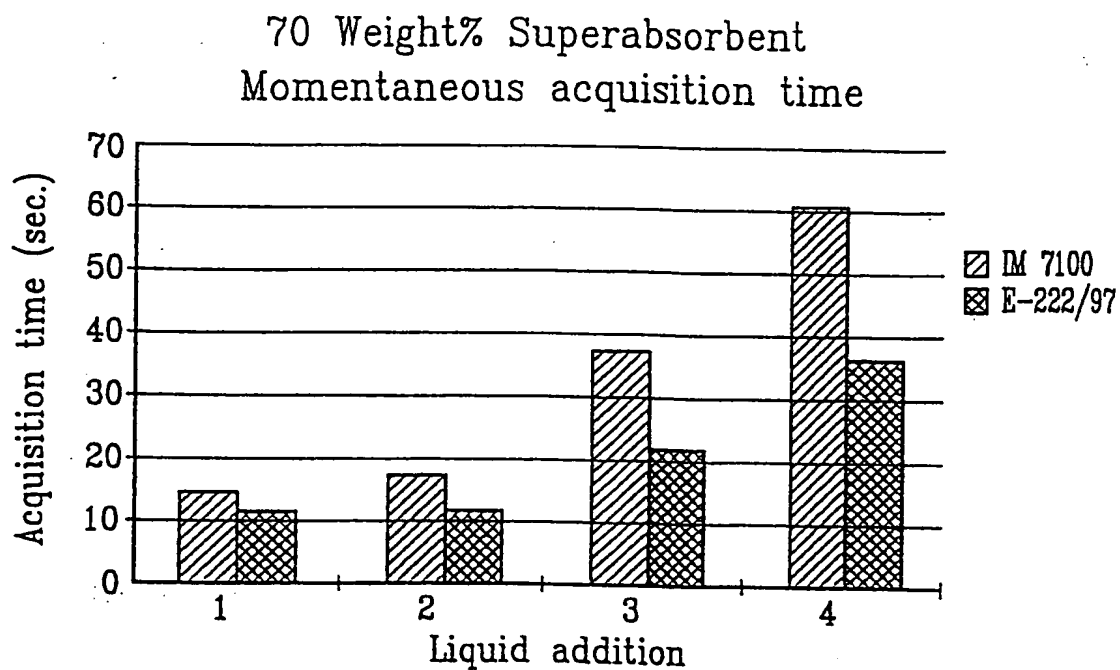


FIG.4b

1  
INTERNATIONAL SEARCH REPORTInternational application No.  
PCT/SE 99/00627

## A. CLASSIFICATION OF SUBJECT MATTER

IPC6: A61L 15/24, A61L 15/60  
According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: A61L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 9325735 A1 (COURTAULDS FIBRES (HOLDINGS) LIMITED), 23 December 1993 (23.12.93), page 8, line 29 - page 9, line 7; page 14, line 17 - line 20, claims 1-3 --	1-13
X	WO 9425519 A1 (CHEMISCHE FABRIK STOCKHAUSEN GMBH), 10 November 1994 (10.11.94), page 1; page 8, line 16 - line 24, claim 2 --	1-13

☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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## C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	<p>EP 0295438 A2 (JOHNSON &amp; JOHNSON PATIENT CARE,            INC), 21 December 1988 (21.12.88)</p> <p style="text-align: center;">--            -----</p>	1-13

**INTERNATIONAL SEARCH REPORT**  
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International application No.

PCT/SE 99/00627

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